National Bureau of Standards

TECHNICAL NEWS BULLETIN

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Rapid Determination of Copper in Iron and Steel

TO CONSERVE the supply of expensive and scarce alloying metals, many of the high-strength structural steels now being prepared for compliance to special or national emergency specifications are designed to utilize the alloy content of the scrap used in the steel making process. This requires rapid methods of analysis so that the composition of the steel bath can be determined and necessary adjustments in composition and treatment can be planned during the melting period.

One of the elements frequently used as an alloying constituent is copper, both to increase the strength of the steel and to improve its resistance to atmospheric corrosion. Though a number of accurate chemical methods are available for the determination of copper in steel, a rapid, one-step procedure specifically designed for the determination of copper in alloy steel has been lacking. This need now appears to have been met by a photometric method developed recently by J. L. Hague, E. D. Brown, and H. A. Bright of the National Bureau of Standards.

In the NBS method, the steel sample is first dissolved in dilute nitric acid. (For high-chromium steels, a mixture of nitric and hydrochloric acids is used.) A sulfuric-phosphoric-perchloric acid mixture is added, and the solution is evaporated until fumes of sulfuric acid are given off. The use of the sulfuric-phosphoric acid mixture provides for the complete solution of tungsten tool steels. The solution is then diluted and, after the addition of citric acid, is made slightly ammoniacal. The sodium salt of ethylenediaminetetraacetic acid is added as a complexing reagent, and sodium diethyldithiocarbamate is also added to form a copper complex. The complex is extracted with butyl acetate, and the butyl acetate extract is washed with diluted sulfuric acid to stabilize the complex. The optical absorbancy of the copper diethyldithiocarbamate complex in the butyl acetate layer is measured in a colorimeter at 560 to 600 millimicrons, and the amount of copper is read from a calibration curve or table. A single determination can be made in approximately 30 minutes, and a set of eight determinations can be completed in about 2 hours.

The reliability and accuracy of the method were checked by making a number of determinations on various NBS standard samples of iron and steel. An accuracy of the order of 0.001 percent of copper was indicated for samples containing less than 0.05 percent of the element. All average values obtained for samples containing from 0.05 to 0.25 percent of copper were within 0.005 percent of the certified value.

The fact that the nickel content of the sample does not interfere in the analysis constitututes an additional advantage of the new procedure, for many of the present-day copper-bearing steels also require the pres-

Determination of the copper content of a steel sample in the NBS analytical laboratory. A colorimeter is being used to measure the optical absorbancy of a copper complex, from which the copper content of the sample is determined by reference to a chart or table.

ence of nickel in the alloy. Stainless and corrosion-resisting steel, nickel-base casting alloys, and tool steel can be analyzed for copper content by the NBS method as easily as carbon steel.

For further details, see Photometric determination of copper in iron and steel with diethyldithiocarbamate, *by John L. Hague, Eric D. Brown, and Harry A. Bright, J. Research NBS* **47,** *380 (1951) RP2265.*

TECHNICAL NEWS BULLETIN

U. S. DEPARTMENT OF COMMERCE

CHARLES SAWYER, *Secretary*

NATIONAL BUREAU OF STANDARDS
A. V. ASTIN, *Acting Director*

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Contents

Hazards From Static Electricity

ELECTROSTATICS, the study of the phenomena of electrically charged particles or bodies, is of fundamental importance to science because many of the basic particles are charged and coulomb forces determine many of the properties of matter. Electrostatic forces are also of increasing industrial importance and have been applied to such processes as ore-separation and large scale spray-painting. Painters, for instance, have found that a tremendous saving is achieved if the object to be painted is charged to several thousand volts.

In other industries, such as air conditioning, the manufacture of explosives, grain and flour storage, textiles, and printing, static electricity, under many circumstances, can be a distinct hazard. In a granary, for instance, the air is usually thick with fine flammable grain-dust particles. Grain sliding down a metal chute can result in an accumulation of static charge that, when it discharges in an electric spark, can easily ignite the fine dust particles and result in a dangerous and expensive fire.

For many years, the National Bureau of Standards has been consulted by other government agencies on problems arising from the hazards associated with static electricity. To meet a growing demand for this information, the Bureau is presently enlarging its research and standardization program, under the direction of F. L. Hermach, for establishing and evaluating methods of measurement and for determining the properties of materials and equipment used to reduce the hazard. The results of one important phase of the program, an investigation of the conductivity of floor coverings, are being utilized to reduce static electricity hazards in hospitals currently under construction and in Federal munition plants.

Specifications for conductive floors and other equipment depend on a determination of the safe limiting resistances between objects. NBS has participated in determining these limits, evaluating various safety factors, and devising suitable methods applicable to field as well as to laboratory testing of flooring and other materials, and is testing and evaluating various types of conductive floorings, coatings, and waxes. This work is conducted only for Federal and other government agencies.

A conductive floor need be only a moderately good conductor of static electricity—a resistance of one million ohms or less is ample for this purpose. Such a flooring provides a safe path over which separated electrostatic charges may be reunited as quickly as they

were separated. It is this natural inclination of the charges to rapidly reunite that causes the hazard, creates the spark, or provokes the shock.

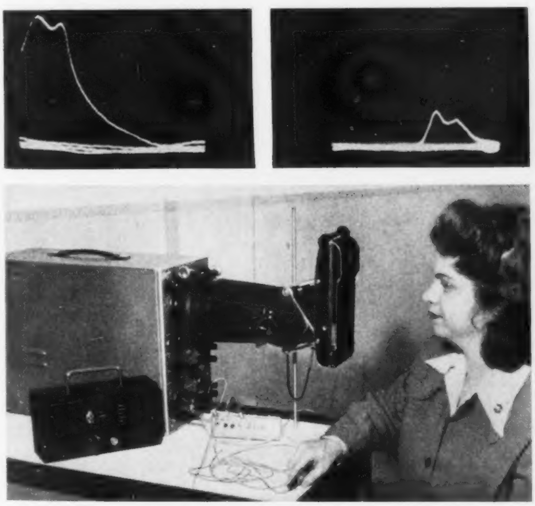
To determine the resistance characteristics of flooring, a sample is subjected to different voltages under varying conditions of humidity and temperature. The measuring procedure is also considered in the evaluation. The flooring resistance is measured with a special resistance-bridge as a function of pertinent variables such as voltage, humidity, and the type of electrode used in making the measurement. One test on a section of terrazzo flooring showed that for a variation of from 30 to 500 volts, the resistance of the sample decreased from about 4.5 to 1.5 megohms. A low resistance flooring—conductive linoleum—changed from almost 3 megohms at 40 volts to less than 10,000 ohms at only 150 volts.

An accompanying phase of these experiments is a determination of the actual safe upper limit of resistance. For example, tests show that voltages as high as 5,000 volts can be developed by such common-everyday-occurrences as getting up from a plastic-covered chair. Oscillographic records, however, indicate that if the resistance between the person and the chair is less than 20 megohms, the voltages will not exceed 300 volts. This is less than the minimum sparking voltage in air and is thus not hazardous.

The magnitude of the electrostatic charges produced by separating objects depends on the material involved, their intimacy of contact, and their surface resistivities. However, many accidents do not arise from these direct effects but rather from the secondary effects resulting when the static electric charges are reunited. For instance, the electric shock experienced when a charged person touches a conducting object, while generally merely annoying, can cause serious damage if the person is precariously balanced on top of a ladder at the time he receives the minor shock. Also, the energies in electrostatic sparks may be large enough to ignite flammable gases or vapors that may be present in an enclosed room. Likewise, the "precipitation static" caused by corona discharges from highly charged aircraft in flight has been found to be often responsible for very serious interruptions to radio communications.

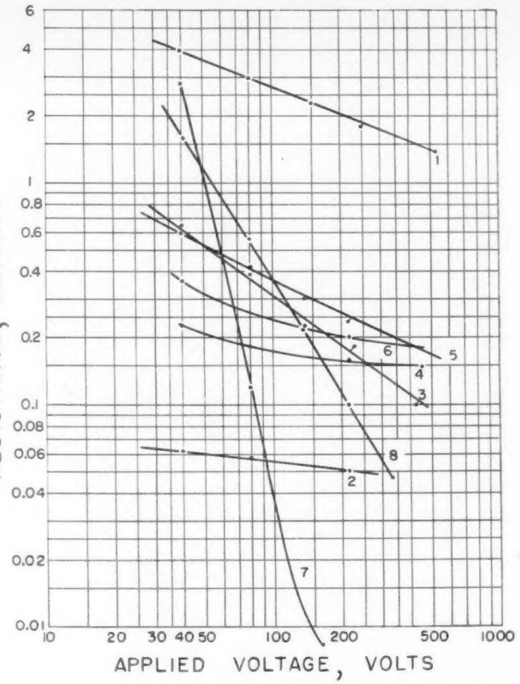
The most serious hazards, those of possible ignition of flammable gases or dusts, can best be mitigated by reducing the flammability or eliminating exposure to the flammable agent. This also reduces the hazard from other sources of ignition, such as arcs from the normal operation of electric equipment or the presence of hot bodies in the hazardous area. These measures, when coupled with the exclusion from the area of materials such as plastics, wool, and rubber, which have high electrical resistivity and "generate" high electrostatic charges, are often sufficient to reduce the hazard to a negligible value.

More elaborate methods of mitigation depend upon reuniting the charges as fast as they are separated in order to keep the voltage across the affected objects low. This can be accomplished by connecting stationary metallic objects to a common ground, by humidification (which provides a film of moisture of moderate conductivity on most objects), or by install-



Left: **Observing the magnitude of static electricity developed by a person rising from a metal chair having a plastic-covered seat. Electrodes are connected to the chair and to the person; the voltage that is developed is recorded on the oscilloscope (left) and at the same time photographed by the attached camera. The inset at the upper left is an oscillogram of the voltage developed when the resistance is high; the one on the right shows the static potential when a moderate conductor is used.** ***Right:*** **Measurement of the contact potential and internal resistivity of a bar sample of flooring. The metal electrodes are connected to the megohm bridge (left), and the bridge reading is an indication of the total resistance and current flowing in the section of the bar between the electrodes. Two other electrodes on the bar (contact made through pools of lead amalgam) are connected to an "inverted" vacuum-tube voltmeter. The reading of this meter is a measure of the internal resistivity of the sample.**

Graph of resistance of floor coverings (in megohms) vs. applied voltage. The plot shows the dependence of electrical resistance upon applied voltage for samples of conductive flooring at room temperatures and 50 percent ambient relative humidity. The tested floor coverings are: terrazzo (1), conductive terrazzo (2) and (3), magnesium-oxychloride (4) and (5), conductive rubber (6), conductive linoleum (7), and conductive plastic (8).



ing a conductive floor to provide electrical contact with the objects that move or rest upon it. The latter is generally considered the safest method where persons or moving objects may separate charges. Thus, in hospital operating rooms, for instance, the personnel normally wear shoes with conductive rubber soles, and the floors are made of conductive material. The separated charges developed in a person by walking, rising from a chair, or removing sheeting are then quickly reunited through the flooring before they can be transferred to another person or object.

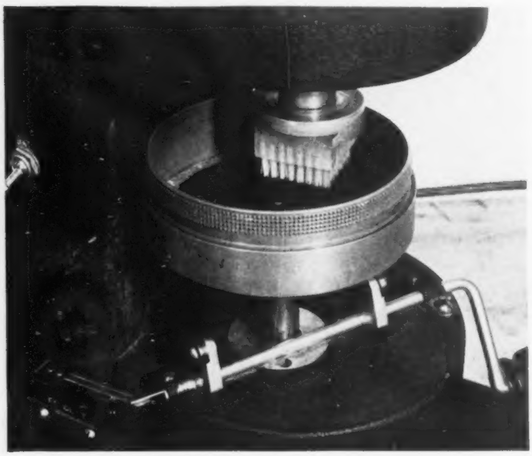
In addition to the consultatory and testing services to other government agencies, members of the NBS staff have also served on a number of committees that have been organized to formulate rules and procedures governing static electricity preventive practices. In 1942, the Bureau published a circular [1] that outlines the problems and suggests solutions that are as timely today as they were 10 years ago. The circular discusses the nature and the origin of the charges of static electricity arising in industrial processes, and explains the various methods of mitigating the hazards that they introduce. It also gives the quantitative basis for an engineering treatment of the phenomena.

[1] *NBS Circular 438*, Static electricity, *available from the Superintendent of Documents, Washington 25, D. C., at 10 cents a copy.*

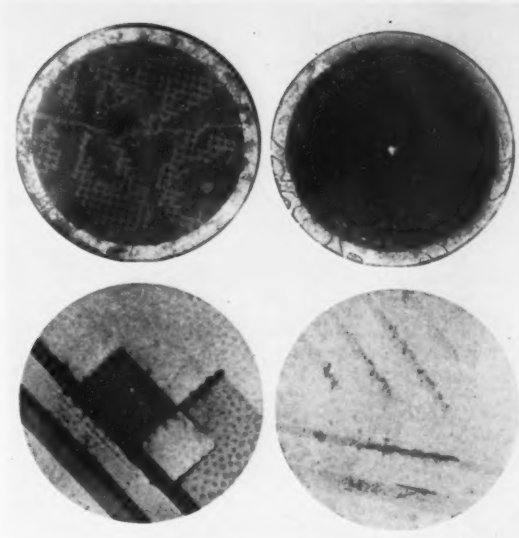
Printed-Enamel Felt-Base Floor Coverings

DESPITE the large volume of commercial production, no generally accepted methods for the evaluation of the properties of printed-enamel felt-base floor covering have been established. To meet this need, methods and equipment have recently been devised by the National Bureau of Standards that provide performance data for such material in good agreement with actual service tests. Developed by George G. Richey, Elizabeth H. McKenna, and Robert B. Hobbs of the NBS Organic and Fibrous Materials Division, the procedure promises to be useful to manufacturers, large purchasers, and others interested in evaluating printed-enamel felt-base floor coverings.

Printed-enamel felt-base floor coverings—known in the trade as "felt-base rugs"—form one of the classes of material called "hard-surface floor coverings," which include linoleum, rubber tile, asphalt tile, and plastic tile and sheet. The annual production of printed-enamel floor coverings in the United States exceeds that of any other hard-surface floor covering. In 1947, for example, production amounted to about



The resistance of printed-enamel felt-base floor coverings to scrubbing with an alkaline soap solution was determined with a Schiefer abrasion machine. The apparatus was modified to accommodate a nylon bristle brush as the abradant and a brass cup for retaining the test specimen and the solution. The brush, cup, and specimen are shown in position on the machine.



NBS specimens of printed-enamel felt-base floor coverings after test for resistance to cleaning compound using the modified Schiefer abrasion machine. Note the areas of localized exposure of the seal coat or felt on some of the floor coverings.

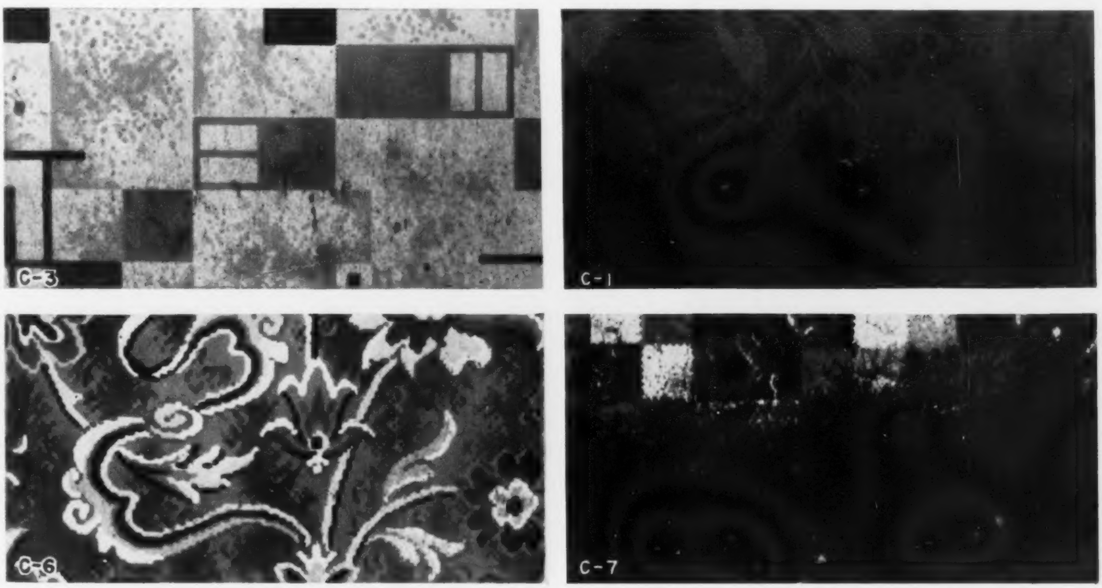
200 million square yards, valued at approximately 80 million dollars at the factory. Printed-enamel coverings are widely used over wood floors in the kitchens of older houses, in summer homes, and in low-cost housing.

This type of floor covering is often mistakenly called "linoleum." Although its superficial appearance resembles that of linoleum, printed-enamel coverings are readily distinguishable by the thickness of the wearing surface. The printed-enamel coverings have a wearing surface of enamel from 0.004 to 0.007 inch thick while the wearing layer of linoleum ranges from 0.030 to 0.085 inch thick.

The NBS study was concerned with such physical and performance properties as the weight, thickness of enamel and seal coat, over-all thickness, resistance to cleaning compound, resistance to kerosene, flexibility, and tearing strength. Of the methods used in this study, those for evaluating tearing strength and resistance to cleaning compound present the most interesting new developments.

A high tearing strength is desirable in order that such coverings may be unrolled and laid on the floor easily without tearing. The NBS procedure for measuring this property consists of an adaptation of the ASTM method for determining the tearing resistance of paper. In particular, the Elmendorf tear tester was modified to double its capacity, and a semicircular specimen is used instead of the rectangular specimen required for paper.

Good resistance to cleaning compound is another highly desirable property, since floor coverings of this type are generally cleaned by scrubbing with a soap solution which is sometimes rather alkaline. In the



Appearance of several specimens of printed-enamel felt-base floor coverings after service tests on a stairway at the Bureau. Ratings were in comparison with each other as A, B, C, or D (best to poorest), on the basis of distinctions of patterns, gloss, resistance to soiling, and general appearance. Specimen *C–6* was rated A; *C–3* as B; *C–1* as C; and *C–7* as D.

NBS test a disk of the floor covering is scrubbed by a nylon bristle brush with a dilute solution of soap and washing soda. The apparatus consists of a Schiefer abrasion machine modified to accommodate the nylon brush and a cup for retaining the solution on the disk. This test procedure has revealed great differences in the durability of presumably comparable materials.

The validity of the test methods was checked by practical service tests of eleven commercial printed-enamel floor coverings. In one test, the corridor floor of the Munitions Building in Washington, D. C., was covered with 5-by-7-foot samples of the materials. After about a year of service, during which the specimens were cleaned 100 times and traversed 800,000 times by pedestrians, the floor coverings were removed and appraised for damage and change in appearance by a group of scientists, manufacturers' representatives, and consumers. In a second test, 10-by-36-inch specimens were cemented to a stairway at the Bureau. After several months of wear, these were also removed and appraised. Both tests revealed the same relative order of durability.

The data obtained from all NBS laboratory tests, with the single exception of the thickness of the seal coat, showed a significant correlation with the service test results. Thus it appears that the proposed NBS tests will be useful in providing a more reliable accelerated evaluation of printed-enamel felt-base floor coverings. The methods and the results obtained by the Bureau may also be used to further improve this type of floor covering.

For further technical details see National Bureau of Standards Building Materials Structures Report, BMS 130, Methods and equipment for testing printed-enamel felt-base floor covering, *by George G. Richey, Elizabeth H. McKenna, and Robert B. Hobbs. See also* Solution of problem of producing uniform abrasion and its application to the testing of textiles, *by Herbert F. Schiefer, J. Research NBS* **39** *(1947) RP1807; and* Improved single unit Schiefer abrasion testing machine, *by Herbert F. Schiefer, Lawrence E. Crean, and John F. Krasny, J. Research NBS* **42** *(1949) RP1988. A summary article on this machine was published in NBS Tech. News Bulletin,* **31**, *No. 6, (June 1947).*

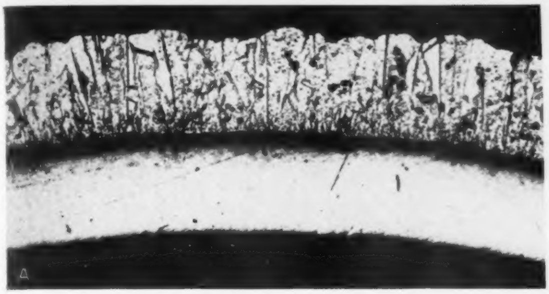
New Aluminum-Plating Process

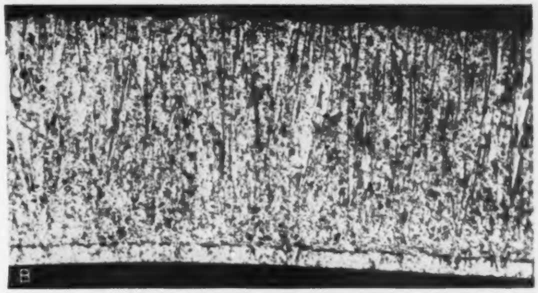
A PRACTICAL process for electrodepositing aluminum at room temperature has recently been developed by D. E. Couch and Abner Brenner of the NBS electrodeposition laboratory. Dense, ductile deposits of the metal are being obtained at the Bureau from a new type of organic plating bath consisting of an ether solution of aluminum chloride and a metal hydride. The new bath is expected to find important application for electroforming articles with close inside tolerances, such as waveguides, and for providing various types of equipment with a thin protective coating of aluminum.

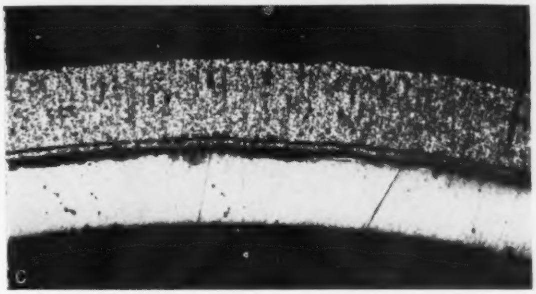
Because aluminum is so far above hydrogen in the electromotive series of the elements, it has never been deposited from aqueous solutions, which always contain some hydrogen in ionized form. Aluminum is usually produced commercially from a bath of fused cryolite and aluminum oxide. However, this process must be carried out at a high temperature, and the metal is obtained in a fused state, unsuitable for electroplating or electroforming. Electrodeposition from nonaqueous solutions has been tried in the past, with some success, but the procedure was too difficult for practical applications, and the deposits were lacking in purity, ductility, and other desirable qualities.

The Bureau is now conducting extensive research in an effort to develop methods for depositing in good physical form unusual metals such as molybdenum, tungsten, titanium, and zirconium. Both fused salts and baths of organic solvents are being investigated. One of the first metals studied was aluminum. The success of the aluminum plating bath, developed in the course of this work, may be expected to give added impetus to the current widespread efforts to obtain other metals in pure form from nonaqueous solutions.

Organic plating bath recently developed by NBS for electrodepositing aluminum at room temperature. To avoid the introduction of moisture, the bath is covered with a closed container through which dry air is passed before the plating operation begins. The bottle in the foreground contains sulfuric acid used as a drying agent.







Photomicrographs of aluminum deposits obtained with the new organic plating bath developed by the Bureau. Deposits obtained at lower current densities (*A*) are characterized by large, columnar crystals. Use of periodic reverse current produces some grain refinement (*B*). Addition of β,β'-dichloroethyl ether to the bath results in a very fine grain structure (*C*). The ductility and hardness of the deposits vary considerably. Deposit *A* is quite ductile while *C* is very brittle. The hardness of deposit *A* is 37 Vickers; of *B*, 67 Vickers; and *C*, 97 Vickers. (× 90).

The aluminum plating bath is prepared at NBS by adding either lithium hydride or lithium aluminum hydride to an ethyl ether solution of anhydrous aluminum chloride. For best results, the ether should be anhydrous and alcohol-free. The concentration of the aluminum chloride is not critical and may vary from 1 to 4 molar. Current densities may be as high as 4 or 5 amperes per square decimeter. However, if thick deposits are desired, the current density should not be greater than 2 amp/dm^2. Because of the high concentration of aluminum chloride in the ether, the bath is not as flammable as would normally be expected.

To prevent the entrance of moisture, the bath is prepared and used in a closed container consisting of a glass plating vessel with a tightly fixed polyethylene lid. Anodes of aluminum rod pass through the lid, and the objects to be plated (cathodes) are introduced and removed through a central hole which is ordinarily kept closed with a rubber stopper. If hermetically sealed, the bath will keep for several weeks; however, under ordinary operating conditions the solution slowly deteriorates and eventually gives streaked and brittle deposits.

Objects electroformed of aluminum by means of the new hydride plating bath developed by NBS. The aluminum was deposited on copper, which was then dissolved out with nitric acid, leaving the aluminum replica.

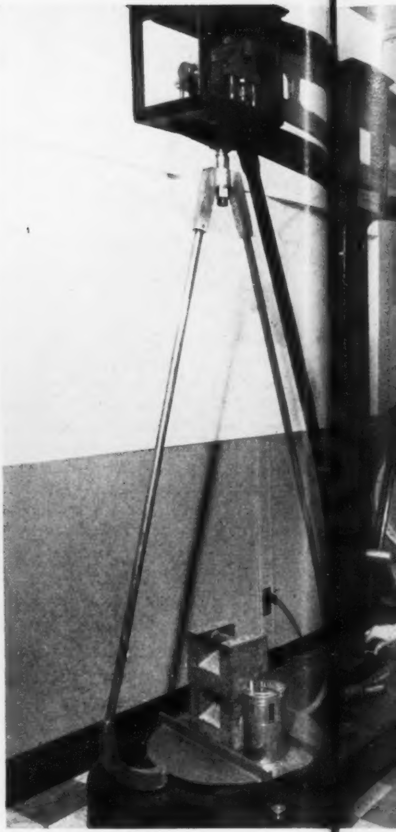
No agitation of the bath is necessary. In fact, a quiescent bath is actually an advantage as the sediment from the anodes settles to the bottom of the vessel, making bagging of the anodes and filtration of the solution unnecessary. The composition of the bath is easily controlled since the only constituent that changes appreciably in concentration during the plating process is the lithium hydride. Occasional additions of lithium hydride increase the life of the bath, but in time the lithium hydride becomes insoluble and the bath can no longer be used.

When sufficient lithium hydride is used, the deposits are white, mat and quite ductile; but if the lithium hydride content is too low (less than 3 or 4 g/l), the deposits become hard, brittle, and grey. Still further reduction of the hydride content produces deposits that are dark and/or stressed and that often crack or peel from the cathode. Ordinarily deposits 0.01 inch or more thick are visibly crystalline, but this effect can be reduced somewhat by adding a small amount of β,β'-dichloroethyl ether. Pitting, which frequently occurs in aqueous baths, is practically nonexistent in the ether bath.

Cathode and anode efficiencies for the process are approximately 100 percent. Deposits 0.05 inch thick have been prepared at the Bureau, and thicker deposits should be possible if the sharp edges of the cathode are shielded to prevent treeing.

For further details, see A hydride bath for the electrodeposition of aluminum, *by Dwight E. Couch and Abner Brenner, J. Electrochem. Soc. 99,* **234,** *(June 1952).*

Studies of Tire Performance



MUCH NEW information on the tread wear, power loss, and other performance characteristics of automotive tires has resulted from an extensive investigation now under way at the Bureau. This program, under the direction of Dr. R. D. Stiehler of the NBS staff, has involved the development of a number of new and improved methods for predicting tire performance. Through the use of statistically planned experiments, it has been possible to determine the effects of many variables not previously isolated. The results are being utilized in the development of improved specifications for Government purchase.

With the introduction of synthetic rubber in the tire industry, large variations in tire quality have developed. At present, the service life of tires of the same size but of different makes ranges from 12,000 to 41,000 miles. As the Federal Government is spending over $50,000,000 this year for automobile tires, large savings should result if tires could be purchased on the basis of performance. To provide the necessary data, the Bureau in 1950 began a large-scale program for the development of tire testing methods under the sponsorship of the General Services Administration. Studies have also been undertaken for the Reconstruction Finance Corporation in connection with current efforts to develop large-size truck and bus tires made with large proportions of synthetic rubber.

Previous NBS work on tire testing methods extends back to about 1920, when investigations were first begun to develop objective procedures for evaluating tires. Because a tire is composed of other materials in addition to rubber, the tests used for rubber compounds were of little use in predicting tire performance and serviceability, and it was necessary to work out new methods of test. These early studies led to the development of the endurance test for tires which is now used in the tire industry and in the current Federal Specification for pneumatic tires. These studies also showed that the variation in power loss among different brands produced during the twenties affected fuel consumption by about 10 percent. Today, with the increasing use of synthetic rubber tires, differences in power loss are much greater, and the need for reliable methods of relating tire characteristics to operational performance has correspondingly increased.

Power-Loss Test

Power loss in tires may be defined as that portion of the effective power produced by the engine which is

Left: Specially equipped vehicle used by NBS for road-testing truck tires with 20-inch rims. The test truck has six-wheel drive and carries an equal known weight on each axle. **Center:** During the test the tires are accurately weighed after each 500-mile period to determine tread loss. The tire is then rotated to a new wheel position in accordance with a statistical design. **Right:** After a tire has been road-tested in each wheel position, the remaining tread is removed by buffing so that the total weight of tread may be determined.

dissipated in the tires as heat. This loss is disadvantageous for two reasons. First, it increases the temperature of the tire. In large tires, the temperature in service may actually become higher than the temperature at which the tire was vulcanized; as a consequence, the textile cords and the rubber rapidly deteriorate and the tire fails. The second disadvantage of a high power loss is the resultant increase in fuel consumption and in the power required of the engine. As most passenger cars have a surplus of power, an increase in the power loss of a passenger tire is not serious except, perhaps, for the increase in fuel consumption. However, in the case of trucks, particularly tractor-trailers with as many as 18 tires, any increase in power loss in the tires may have a pronounced effect on both vehicle performance and fuel consumption. The effect is particularly noticeable on hills, where the magnitude of the power loss may determine whether shifting of gears is necessary.

Recently the Bureau has constructed a machine of special design which measures the amount of power lost in a tire when load, tire pressure, speed, tractive effort, and cornering are independently varied. The machine employs two dynamometers. One measures the power required to drive the tire while the other measures the tractive effort, or the power transmitted by the tire to a steel drum. The power loss is the difference between these two measurements after correction has been made for windage and for power loss in the bearings due to friction.

Power loss is found to be influenced by both composition and design of the tire. Because of the large increase in power loss when synthetic rubber is substituted for the natural product, large truck and bus tires at present must be made in large part from natural rubber. However, efforts are being made to develop more elastic types of synthetic rubber that will have lower power loss.

Carcass Tests

The flexing of a tire in service, particularly at elevated temperatures, causes fatigue of the cords and rubber. Both composition and design affect the endurance of a tire but not necessarily in the same way as they affect power loss. Thus, while a tire with a low power loss may not have good fatigue resistance, it is generally true that a tire with a high power loss will have poor fatigue resistance. This is due to the rapid deterioration that takes place at the high running

The National Bureau of Standards tire test fleet. The three trucks (left) with six-wheel drive are used in determining the tread life of truck tires having 20-inch rims. The other four trucks, equipped with four-wheel drive, are used in testing passenger and light truck tires.

temperature, which may be considerably above the boiling point of water.

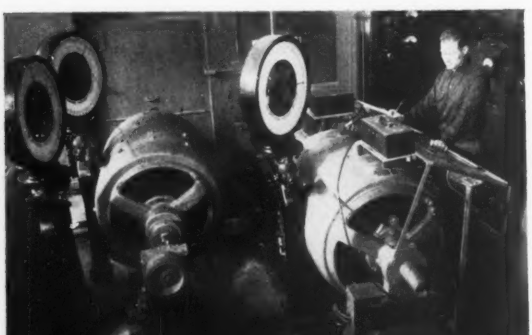
Endurance or fatigue is measured at NBS by running the tire against a steel drum continuously for several days. This test is similar to the power-loss test, but an ordinary motor is used instead of a dynamometer as the motive force, and the load on the tire is increased at intervals. The process is continued to failure for research studies but is discontinued after a specified time in testing for compliance with the Federal Specification. Running temperatures may also be measured during the test. A spread in running temperature of 68 Fahrenheit degrees has been found in tests on different brands of the same size of tire.

The energy required to rupture a tire is a measure of the strength of the tire and its bruise resistance. These properties are determined in a static test in which the rupturing force and deflection are measured at the moment a cylindrical plunger with a rounded end breaks through the crown of the tire. From these measurements, the energy of rupture is computed. If the test is made on a tire that has been in service or one that has undergone the endurance test, the energy for rupture may be much lower than that of an unused tire. The difference between the rupture energies before and after use can thus serve as a measure of the deterioration that has taken place. In this way it has been found that tires made with cotton cords deteriorate much faster than those made with rayon or other synthetic fibers. An accelerated aging test based on the plunger test is now under development at the Bureau.

Tread Tests

When a tire is poorly designed or when inferior tread compound is used, cuts or cracks in the tread grooves may rapidly increase with length in service and cause premature failure. This condition is determined at NBS in a test which is conducted along with the endurance test. Predetermined numbers and lengths of cuts are placed in the tread grooves before the endurance test, and the lengths of the cuts are measured after the test to determine the cut growth.

The present test for tread life is based on investigations carried out during World War II. When this country's normal supply of natural rubber was cut off, both civilian and military transportation were seriously threatened. Many substitute materials were suggested for recapping tires, and the more promising of these were submitted to the National Bureau of Standards for evaluation. As there was then no standard test for determination of tread life, a search was made for



Dynamometer installation at NBS for studying power loss in tires. The tire under test is contained in the temperature-controlled enclosure in the background. The dynamometer at right measures the power required to drive the tire while the other dynamometer (left) measures the power transmitted by the tire to a steel drum.

NBS determines the strength and bruise resistance of tires by measuring the force and deflection at the moment a cylindrical plunger breaks through the tire.

a rapid and reliable method for measuring the rate of wear of tire treads.

The tire industry has been measuring this property by the decrease in depth of the tread grooves. However, the decrease in groove depth does not take place at a constant rate but becomes slower with increasing mileage because of the change in the area of tread in contact with the roadway. Thus, to obtain reliable results from the depth measurements, it is necessary to wear the tires smooth in long and expensive road tests. At NBS it was found that, unlike the decrease in groove depth, the rate of loss in weight of a tire is essentially constant throughout the life of the tread. This finding permitted much shorter and less expensive road tests since the rate of tread wear could then be determined by differential weighings in a small fraction of the miles required by the depth method. The shorter road test which the Bureau developed as a result of this work forms the basis for the tread-life test in the interim Federal Specification now in effect.

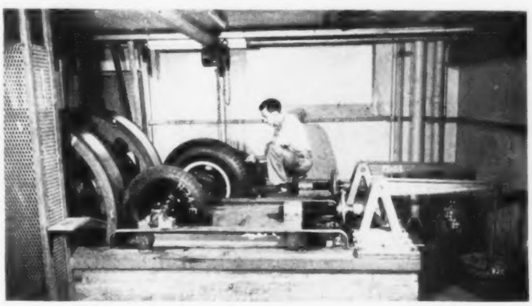
Because of the greater number of variables involved, road tests are much more difficult to control than laboratory tests. Recently the Bureau has developed an improved method of test for tread wear which utilizes the weight-loss method but also employs a statistical design, based on the latin square, to compensate for differences in the treatment received by each tire. In this way, differences in wear due to the use of the tires on different test vehicles or in different wheel positions are taken into account. Because of changing climatic and road conditions, tires are always evaluated relative to those included in the same road test.

The Bureau is now conducting road tests with a fleet of seven trucks. Four of the trucks are equipped with four-wheel drive; the others have six-wheel drive. The vehicles with four-wheel drive are used in testing passenger and light truck tires having 15- and 16-inch rims while the other trucks are used for truck tires having 20-inch rims. At the end of each 500-mile period, the tires are washed, carefully weighed, and transferred to a new wheel position in accordance with a 4 by 4 or 3 by 3 latin square design. After each tire has been tested in each wheel position, the remaining tread design is removed by buffing to determine the total weight of tread. Tread life is then calculated from this weight in combination with the rate of wear determined in the road test. Much new information is being obtained in this way. It has been found that the tread life of different brands of tires may vary by a factor of 3 or more. It has also been found that rate of wear is influenced by both the type of rubber and the type of "black" used in the rubber, but that power loss is influenced more by type of rubber than by type of black.

Fundamental Studies

In addition to investigations of tire performance and the development of improved methods for evaluating tires, the Bureau is conducting other studies of a more fundamental nature that directly concern the use of rubber in transportation. For example, a broad program is under way on the dynamic properties of rubber. The viscous properties of rubber result in power loss in tires, and this energy loss is dependent on the frequency of vibration of the tire. In the Bureau's study, the viscous and elastic properties of rubber are being determined as a function of frequency so that rubber may be used more intelligently in tires and other resilient products. Other investigations, which deal with the vulcanization and stress-strain properties of rubber and the testing and grading of natural rubber, provide information of importance in controlling the quality of rubber used in tires.

One research program, of vital concern to the armed services, deals with the properties of rubber at low temperatures. Tires, belting, or other rubber articles



Tire endurance is measured by running the tire against a steel drum until failure occurs. Before the start of the test, cuts are made in the tread grooves. The increases in the lengths of the cuts during the test provide a measure of the tendency of cuts or cracks in the grooves to increase during service.

for use in arctic regions or at high altitudes must withstand low temperatures without loss of their characteristic rubber-like properties. Yet most ordinary rubbers lose their elasticity around $-50°$ C ($-60°$ F). Pioneering investigations seeking useful low-temperature rubbers were made at NBS before World War II. These studies are now being continued on a more extensive scale, and much new, significant information has already been obtained.

See also Some factors influencing the road wear of tires, by *R. D. Stiehler, M. N. Steel, and J. Mandel, Trans. Inst. Rubber Ind.* **27,** *298 (1951) and* Tread wear of tires, *by John Mandel, Mary N. Steel, and Robert D. Stiehler, Ind. and Eng. Chem.* **43,** *2901 (1951).*

Effect of Moisture and Ozone on Cotton Textiles

WHEN water evaporates from cotton cloth, a brown line is often observed at the boundary between the wet and dry areas. Recent studies at NBS indicate that this "brown-line effect" may contribute significantly to the degradation of cotton during production, processing, and use. From the data, it appears that continuous evaporation of water at the boundary line causes the cellulose in the cloth to oxidize at ordinary temperatures, with consequent weakening of the material. These results were obtained by H. Bogaty, K. S. Campbell, and W. D. Appel of the Bureau's textile laboratory in an experimental investigation of the effects of moisture and ozone on cotton textiles.

The progressive deterioration of cotton cloth when exposed to the weather in tarpaulins, tents, awnings, and various items of military equipment has long been a problem. Sunlight, mildew, and acid-containing industrial atmospheres are important causes of the deterioration which have been studied extensively. However, complete data on the effect of moisture and ozone have been lacking. As part of a broad program of research on the degradation of cellulose, sponsored at NBS by the Office of the Quartermaster General, the Bureau undertook to isolate these two factors and to determine the nature and relative importance of their contributions to textile weathering.

Effect of Moisture

The Bureau's study of the effect of moisture was concentrated on the boundary between wet and dry areas because weakening is especially rapid there. Such boundaries form when water rises in cotton by capillary action or when a drop of water falls on the dry cloth. The brown line is frequently seen on window curtains that have been wet at one end by rain or on cotton that has dried faster at one place than another during laundering or processing.

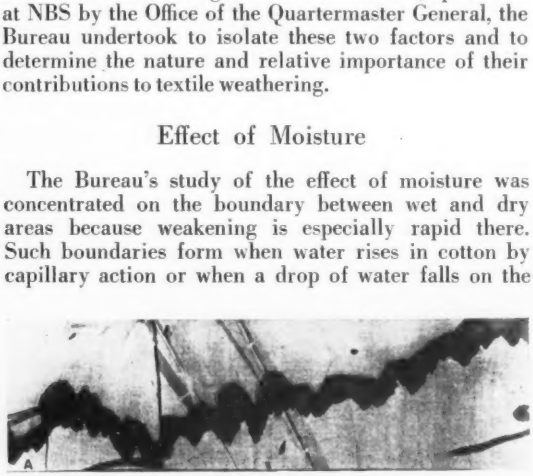
Brown-line experiments, in which one end of a strip of fabric was kept immersed in water under controlled conditions, were carried out at room temperature. In general, several hours were sufficient to produce a distinct yellowish-brown color at the wet-dry interface. The brown line could be produced in viscose rayon, filter paper, scoured cotton, and bleached cotton. It was found to consist of a fluorescent, water-soluble material.

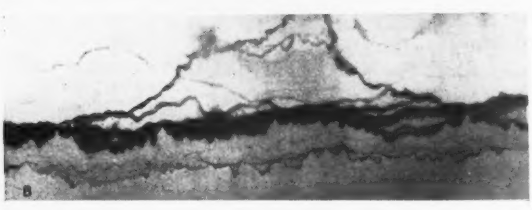
The rate of production of the brown material was remarkably constant. A commercially bleached, sizing-free, alcohol-extracted print cloth was found to produce about 2 mg per day per linear foot of interface. This rate was the same whether the experiments were run in the dark, in the diffuse daylight of the laboratory, or in the light from a tungsten incandescent lamp.

Other experiments showed that the water-soluble brown material could be extracted from fabrics subjected to other types of degradative processes. For example, significant amounts of the brown fluorescent substance were produced in cotton cloth weathered out of doors for six months, or heated in the dark for 10 days, or exposed to radiation from a carbon arc for six hours.

To investigate the possibility that the brown substance might have already existed in the fabric and subsequently concentrated at the interface, a sample of cloth was alternately exposed in brown-line experiments and water-extracted over a period of eight months. As no decrease in rate of formation of the extractable brown substance was observed, it was concluded that the brown material is formed at the interface by a chemical change in the cellulose. The possibility that the brown line could be produced by iron or other inorganic constituents of the material was ruled out by the results of spectroscopic analyses.

Further chemical study of the brown-line substance indicated that oxycellulose of the mildly reducing acidic type is formed. The oxidation appears to occur





The "brown-line effect," caused by the evaporation of moisture from cotton textiles. *A:* brown-line region on a window curtain subjected to frequent wetting by rain over a period of three years. *B:* similar brown-line region on a sample of purified airplane cloth exposed to evaporating moisture in the Bureau's textile laboratory for three months.

in part at the primary hydroxyl group of the cellulose molecule. Polyglucuronic acid is first formed, after which the chain breaks into soluble, low molecular weight fractions.

Other workers have found that small amounts of a soluble reducing substance are obtained by repeated extraction and drying of pure cellulose. This reaction may be similar to the brown-line effect. Numerous other phenomena of industrial importance—such as the unexplained spots observed in paper chromatography and the brownish areas which appear on goods during shut-downs in paper mills and textile finishing plants—may well be caused by a similar reaction.

Action of Ozone

Ozone is formed from oxygen in the upper atmosphere by ultraviolet radiation. Although the amount formed is quite small—0.06 part or less per million parts of air—atmospheric ozone has been found to have significant effects on materials. For example, it causes serious deterioration in rubber under tension.

In the NBS ozone experiments, samples of cotton cloth, both wet and dry, were exposed for varying periods to air containing ozone in known amounts. The dry cloth deteriorated very little or not at all, even in long exposures; but when the cloth was kept moist, substantial deterioration was observed.

At high ozone concentrations deterioration was rather rapid: Three or four days of exposure to air containing 160 parts per million of ozone resulted in a very considerable loss of strength. However, when the ozone concentration was reduced to 0.06 part per million, deterioration proceeded very slowly.

Moist cotton duck exposed to the lower concentration for 50 days lost only 20 percent of its breaking strength. Under ordinary conditions of outdoor use, a concentration of this magnitude could hardly be expected to occur along with the requisite wetness of cloth more than 10 percent of the time. At this rate, 500 days of outdoor exposure would be required for the duck to lose 20 percent of its strength through the action of ozone alone. In practice, unprotected cotton fabrics deteriorate much more rapidly. Thus, the NBS study indicates that the contribution of ozone to deterioration of cotton in weather exposure must be relatively small. It would appear that the effects of heat and sunlight are much greater than that of ozone.

For further technical details, see Some observations on the evaporation of water from cellulose, *by Herman Bogaty, Kenneth S. Campbell, and William D. Appel, Textile Research Journal* **22**, *75 (1952); and* The oxidation of cellulose by ozone in small concentrations, *by the same authors, Textile Research Journal* **22**, *81 (1952).*

Fire Tests of Escalator Shutters

UNPROTECTED escalator openings in department stores and other large multistoried buildings may allow a fire to spread with great rapidity. To reduce this type of hazard, the Building Exits Code of the National Fire Protection Association recommends that each moving stairway above street level be equipped with a rolling shutter that will automatically close the top of the stairway when fire or smoke is detected in its vicinity. Automatic shutters of this kind have been installed in many buildings, but definite information on their effectiveness has been lacking. Recently N. D. Mitchell, E. W. Bender, and J. V. Ryan of the National Bureau of Standards completed fire tests of escalator shutters submitted by manufacturers, providing information of interest to architects and builders, as well as regulatory bodies concerned with specifications or building-code requirements.

In general, the shutters that were tested consisted of galvanized steel slats which travel on rollers in steel guide rails. Automatic closure may be obtained by any standard fire- or smoke-sensitive system.

Three fire tests were made: an exploratory test to determine the adequacy of the shutter and guides alone, and two full-scale tests in which the supporting frames and runways were used. The shutters were installed over a floor-test furnace, and fires were applied to the underside of the installation. Temperatures were measured by means of chromel-alumel thermocouples protected by porcelain insulators and encased in wrought-iron pipes sealed at one end. The furnace temperature was raised to 1700° F in one hour, and to 1925° F in three hours in accordance with the ASA

Fire shutter for moving stairway, mounted at the top of a test furnace at NBS. The iron pipes (right foreground) contain thermocouples for measurement of temperatures in the furnace when a flame is applied to the underside of the shutter.

standard method for fire tests. The tests were discontinued at the end of three hours.

In each of the tests the shutters sagged, the guides were somewhat deformed, and the rollers were damaged by burning and loss of lubrication. However, as the fire did not spread up through the shutters, they were considered to have successfully performed their function. Retraction after the fire was difficult, but this did not affect the main function of the shutter. As a result of the tests, design changes have been recommended, particularly to allow for thermal expansion in the guide rails.

For further technical details, see Fire resistance of shutters for moving-stairway openings, *by Nolan D. Mitchell, Edward W. Bender, and James V. Ryan, NBS Building Materials and Structures Report 129, available from U. S. Government Printing Office, Washington 25, D. C., at a cost of 10 cents.*

Pigmentation in Animal Fibers

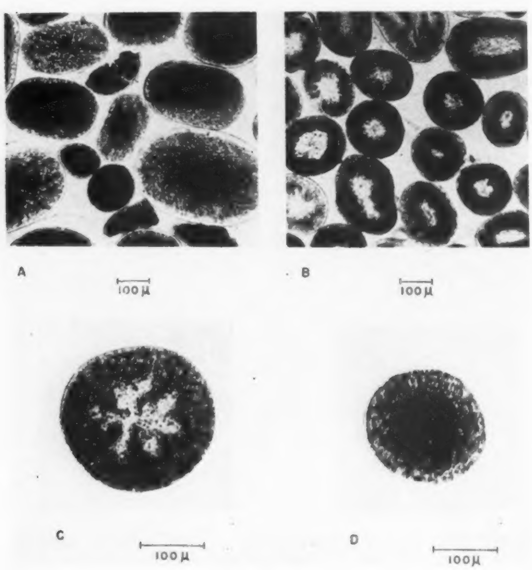
A RAPID, reliable procedure for differentiating between hog bristle and other animal fibers, such as horsehair, has been devised by Dr. S. B. Newman of the Bureau. The NBS procedure, which is based on the difference in the distribution of pigment in the fiber cross sections, provides a more adequate basis for judging paint brushes and other bristle products than the rule-of-thumb methods that are commonly used in the trade.

In determining the fiber content of brushes and samples of bristle, the so-called "eye and feel" tests, based on the tester's previous experience in handling and dealing with the fibers, are most often employed. As the typical bristle is a flagged, tapered fiber, while horsehair is neither flagged nor tapered, it is not too difficult to distinguish a large percentage of the bristles from horsehair in this way. However, in every sample of bristle, particularly in the finer grades, there are many fibers which lack flags and in which the taper is negligible. For this reason, a more objective method has been needed for definite differentiation between these two fibers.

The use of differences in pigment distribution to differentiate between bristle and horsehair had previously been suggested. However, this method appeared to have several possible drawbacks, such as the difficulty of preparing microscopic specimens and the possibility of variations in pigmentation at different levels in the fiber, in different types of hair from the same animal, and in animals from different geographical locations. The Bureau therefore began an extensive microscopic study of bristle and horsehair. Samples were collected from a wide variety of commercial sources, from living animals, and from museum specimens representing a broad geographical distribution.

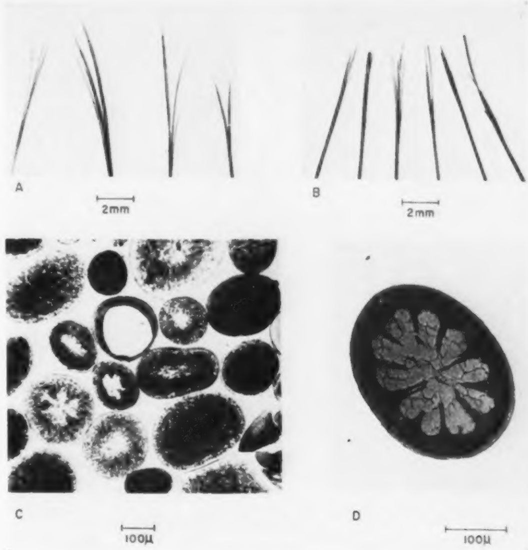
Samples of brushes containing 200 or more fibers were sectioned without difficulty by means of a conventional microtome. Individual fibers were embedded in methacrylate polymer before sectioning, using a process developed by NBS in 1949. The sections were mounted and the pigment distribution studied. It was found that in general the pigment in hog bristle is more dense at the center of the cross section and becomes more dilute at the periphery, whereas in horsehair the pigment is concentrated toward the periphery and exhibits little or no natural color in the central portion. These basic differences were found in all fibers regardless of the geographic origin of the animal, the particular hair type, or the part of the animal body from which the hair was obtained. Differences in the medullation of the two fibers also aid in their analysis. Many of the bristles are hollow while the horse fibers possess a cellular medulla.

Further studies showed that the pigmentation pattern of the fiber is a characteristic of the particular taxonomic family to which the animal belongs. Thus, other species of the horse family (Equidae) were found to exhibit the same pigmentation pattern as the domestic horse while other species of the pig family (Suidae) resembled the domestic pig in fiber pigmentation. On the other hand, when hair from the peccary, a member of a family (Tayassuidae) closely related to the pig family was tested, it was found to have a completely different type of cross section.



Differences in fiber cross section form the basis of the procedure devised by the Bureau for differentiating between bristle and horsehair. This pigment in hog bristle (*A, D*) is more dense at the center of the cross section, whereas in horsehair (*B, C*) the pigment is concentrated toward the periphery.

Differences between naturally flagged bristle (*A*) and artificially flagged horsehair (*B*) are readily detectable under the microscope. *C:* Cross section of fibers from an adulterated brush, showing horsehair (light centers) mixed in with bristles (dark centers). *D:* Hair from the peccary, a member of the family Tayassuidae.



Artificially flagged horsehair is often found in bristle brushes. Synthetic flags, as would be expected, were found on fibers with little or no taper. The flagging occurred practically at one point on the length of the fiber, and many branches, often a dozen or more, were found in the flag. At the point where the flag begins to form, the hair seemed to increase in diameter. Natural flags contained fewer branches, and these divided at different levels from the main trunk.

From the NBS studies, it was concluded that differences in pigmentation pattern may be used to differentiate between horsehair and bristle of all types except those that are naturally white or bleached. As the quantity of unpigmented fiber used in commercial brushes is relatively small, the pigmentation method provides a firm basis for differentiation between bristle and horsehair in commercial products. Development of test methods for differentiating the colorless fibers is now under way at the Bureau.

For further technical details, see Pigmentation in bristle and horsehair, *by Sanford B. Newman, J. Research NBS* **48**, *4 (1952) RP 2315; also see* NBS method for microsectioning, *NBS Technical News Bulletin* **33,** *94 (Aug. 1949).*

New Radio Propagation Disturbance Warnings

BEGINNING July 1, 1952, the Bureau will broadcast new short wave radio disturbance forecasts via the NBS standard frequency broadcasting station WWV. This new service will replace the radio disturbance warning notices that have been transmitted by WWV since 1946. The broadcasts will tell users of radio transmission paths over the North Atlantic the condition of the ionosphere at the time of the announcement and also how good or bad communication conditions are expected to be for the next 12 hours.

The NBS radio disturbance forecasts, prepared four times daily, will be transmitted in Morse code twice each hour—19½ and 49½ minutes past the hour—on WWV standard frequencies of 2.5, 5, 10, 15, 20, and 25 Mc, as was done prior to July 1. As in the past, the notices will include a letter indicating present radio reception conditions. *However, the new notices will also contain a digit indicating the expected quality of future reception.* As before, the letters used will be "N", "U", and "W", signifying that radio propagation conditions are normal, unsettled, or disturbed, respectively. The digit will be the forecast of expected quality of transmitting conditions on the NBS-CRPL scale of 1 (impossible) to 9 (excellent).

If, for example, propagation conditions at the time the forecast is made are normal but are expected to be only "fair to poor" within the next 12 hours, the forecast would be broadcast as N4 in Morse code, repeated five times, i. e., "N4, N4, N4, N4, N4."

Digit (forecast)	Propagation condition	Letter (current)
1	Impossible	W
2	Very Poor	W
3	Poor	W
4	Fair to Poor	W
5	Fair	U
6	Fair to Good	N
7	Good	N
8	Very Good	N
9	Excellent	N

The NBS forecasts are based on information obtained from a world-wide network of geophysical and solar observatories. Data on the development of sunspots, solar eruptions, and other activities of the sun are funneled into the NBS Central Radio Propagation Laboratory in Washington, D. C. Radio soundings of the upper atmosphere, short-wave reception data, and similar information are also readily available. Trained forecasters digest the information and formulate the predictions. The forecasts are issued by NBS regularly each day at 0500, 1130, 1700 and 2300 UT (Universal Time). Each forecast statement will be broadcast by WWV for a period of about six hours—until the next forecast is issued. Thus the forecast prepared at 1700 UT will be first broadcast at 1719½ and then at half-hourly intervals through 2249½. The broadcast at 2319½ will then carry the next disturbance forecast issued at 2300 UT.

The letter portion of the forecast statement, describing the quality of radio propagation conditions, is

valid only for the North Atlantic transmission path at the time the forecast is issued from NBS. The digit portion is a forecast of the average quality of communication conditions along these paths in the 12-hour period beginning at 0000, 0600, 1200, or 1800 UT—about an hour after the time at which the letter describes the conditions. For example, a forecast statement of "W5" issued at 0500 UT means that at 0500 the conditions across the North Atlantic path were disturbed and that in the period 0600–1800 the average of conditions is expected to improve to quality 5 (fair).

The new NBS radio disturbance forecasts refer only to North Atlantic paths, such as Washington to London or New York to Berlin. The forecasters assume that the most suitable radio frequencies for communications are available and in use along these paths. Because of this assumption, their notices must be interpreted on a relative scale in terms of experience on each radio circuit in use. It is impossible to rate conditions on an absolute scale because the varied effects of transmitter power, type of communications traffic and procedure, antennas and receivers prevent an evaluation that will be valid for all systems and all circuits. One purpose of broadcasting both a description and a forecast is to show more clearly whether propagation conditions are expected to deteriorate or improve in the 12-hour period.

For the past 18 months, the NBS Radio Warning Service has been making continuous 24-hour daily studies of the North Atlantic circuits by specialized techniques. The new disturbance information to be transmitted by WWV is one of the results of this investigation. Other radio disturbance forecasts which NBS has supplied regularly for almost ten years are forecasts of propagation conditions 1 to 25 days in advance and daily 24-hour forecasts. Neither of these services are broadcast by WWV but are distributed by airmail, telephone, and telegraph. Similar forecasting services are provided for North Pacific circuits by the NBS North Pacific Radio Warning Service at Anchorage, Alaska.

Publications of the National Bureau of Standards

Periodicals

Journal of Research of the National Bureau of Standards, volume **48**, number 5, May 1952 (RP2322 to RP2328, incl.)

Technical News Bulletin, volume **36**, number 5, May 1952, 10 cents.

CRPL–D93. Basic Radio Propagation Predictions for August 1952. Three months in advance. Issued May 1952. 10 cents.

Research Papers

Reprints from Journal of Research, volume **48**, *No. 4, April 1952.*

RP2312. Pressure-humidity apparatus. Arnold Wexler and Raymond D. Daniels, Jr.

RP2313. Heats of copolymerization of butadiene and styrene from measurements of heats of combustion. Raymond A. Nelson, Ralph S. Jessup, and Donald E. Roberts.

RP2314. Infrared spectra of methanol, ethanol, and n-propanol. Earle K. Plyler.

RP2315. Pigmentation in bristle and horsehair. Sanford B. Newman.

RP2316. The system beryllia-alumina-titania: Phase relations and general physical properties of three-component porcelains. S. M. Lang, C. L. Fillmore, and L. H. Maxwell.

RP2317. Effect of temperature on the tensile properties of high-purity nickel. William D. Jenkins and Thomas G. Digges.

RP2318. Thiophosphation of 2-methyl-2-nitro-1-propanol and the preparation of monothiophosphoric acid. J. V. Karabinos, R. A. Paulson, and W. H. Smith.

RP2319. Surface changes in an original and an activated bentonite. Juan de Dios Lopez-Gonsalez and Victor R. Deitz.

RP2320. Arc spectra of gallium, indium, and thallium. William F. Meggers and Robert J. Murphy.

RP2321. On the truncation error in the solution of Laplace's equation by finite differences. Wolfgang Wasow.

Building Materials and Structures Report

BMS127. Effect of aging on the soundness of regularly hydrated dolomitic lime putties. Lansing S. Wells, Walter F. Clarke, and Ernest M. Levin. 15 cents.

BMS130. Methods and equipment for testing printed-enamel felt-base floor covering. George G. Richey, Elizabeth H. McKenna, and Robert B. Hobbs. 15 cents.

Handbooks

H51. Radiological monitoring methods and instruments. 15 cents.

Miscellaneous Publications

M203. Index to the reports of the National Conference on Weights and Measures. From the first to the thirty-sixth 1905 to 1951. William S. Bussey and Malcolm W. Jensen. 20 cents.

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A simplified method for measuring the attenuation of balanced transmission lines. R. C. Powell. Proc. Nat. Elec. conf. (852 East Eighty-third Street, Chicago 19, Illinois) **7**, 287 (February 1952).

Absence of spontaneous emission of neutrons from samarium. S. D. Chatterjee and R. N. Olcott. Phys. Rev. (57 East Fifty-fifth Street, New York 22, N. Y.) **85**, 147 (January 1, 1952).

The sea-level latitude variation of fast cosmic-ray neutrons. P. S. Gill and L. F. Curtiss. Phys. Rev. (57 East Fifty-fifth Street, New York 22, N. Y.) **84**, 591 (November 1, 1951).

Electron-tube curve generator. M. L. Kuder. Electronics (330 West 42d St., New York 18, N. Y.) **25**, No. 3, 118 (March 1952).

Precise measurements in the infrared spectrum of carbon monoxide. Earle K. Plyler, W. S. Benedict, (NBS), and Shirleigh Silverman (APL, The Johns Hopkins University). J. Chem. Phys. (57 East Fifty-fifth Street, New York 22, N. Y.) **20**, 175 (January 1952).

Kinetics of OH radicals from flame emission spectra. IV. A study of the hydrogen-oxygen flame. H. P. Broida and K. E. Shuler. J. Chem. Phys. (57 East Fifty-fifth Street, New York 22, N. Y.) **20**, 168 (January 1952).

Crystal Chemistry Symposium. Fred Ordway. Ceramic Age (421 Parker St., Newark 4, N. J.) **58**, No. 5, 28 (November 1951).

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